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CHARACTERIZATION OF AS-GROWN DISLOCATION STRUCTURE IN NIOBIUM B--ETC(U)
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CHARACTERIZATION OF AS-GROWN DISLOCATION STRUCTURE IN NIOBIUM BY X-RAY DIFFRACTION TOPOGRAPHY.

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CHARACTERIZATION OF AS-GROWN DISLOCATION STRUCTURE IN NIOBIUM BY X-RAY DIFFRACTION TOPOGRAPHY

by

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The behavior of dislocations in b.c.c. metals has been extensively examined using relatively macroscopic methods, such as deformation studies and etch pitting, as well as with microscopic TEM methods. While these studies have led to significant increases in the understanding of dislocation behavior, the need remains for a method for studying the microscopic behavior of dislocations in relatively thick specimens. One such method, x-ray diffraction topography, has not been extensively applied due to the relatively high x-ray absorption of many of the metals of interest, such as niobium, and the consequent need to prepare highly perfect thin crystals. In the present note we report the preparation of such niobium crystals and the use of Lang topography to characterize their dislocation structures.

Single crystal niobium specimens of thickness suitable for transmission topographic studies were grown by recrystallization of a heavily deformed polycrystalline ribbon of niobium. The procedure consisted of resistance heating at 2200K and 2.6×10^{-8} Pa for times on the order of an hour. It should be noted, however, that due to outgassing of the niobium, protracted annealing was required to obtain this pressure. During this anneal exaggerated grain growth (secondary recrystallization) occurred which was apparently driven by the surface energy of the niobium-vapor interface. Grains were produced having surface area of several cm^2 and having <110> approximately normal to the surface. Rapid cooling, achieved by cessation of the heating current, was necessary in order to minimize severe oxidation at

intermediate temperatures. In the particular crystal described here, the recrystallization anneal was followed by equilibration with 2.9 x 10^{-3} Pa of pure N_2 gas to introduce nitrogen solutes into the niobium. The nitrogen solutes served to reduce the sensitivity of the crystal to strains. The thickness of the crystal reported in the present note is 76 μm, and the crystal has a surface normal about five degrees from $[\bar{1}10]$. Single crystals grown by this method have total dislocation densities of 10^3 cm/cm^3 or less.

A conventional Lang camera using MoKα radiation from a microfocus generator operating at 50 kV and 3.8 mA was used for the topography. Ilford L4 nuclear emulsions with a thickness of 50 μm recorded the topographs. Because $\mu t \sim 1$, where μ is the linear absorption coefficient and t is the crystal thickness, kinematical contrast is expected to predominate. The Burgers vectors of some of the dislocations in a network were determined using the following criteria. Neglecting elastic anisotropy, a dislocation will be completely invisible if $\underline{g} \cdot \underline{b} = 0$ and $\underline{g} \cdot \underline{b} \times \underline{u} = 0$, where $\underline{g}$ is the diffraction vector, $\underline{b}$ is the Burgers vector and $\underline{u}$ is the dislocation line vector. Residual contrast may occur if $\underline{g} \cdot \underline{b} = 0$, but $\underline{g} \cdot \underline{b} \times \underline{u} \neq 0$; if both conditions hold but the material is as elastically anisotropic as niobium; or if there is a segregation of impurity atoms to the dislocations. An additional requirement for networks is that the sum of Burgers vectors of dislocations meeting at a node must be zero.

Projection topographs were taken with the following

diffraction vectors: 200, 020, $00\bar{2}$, 110, 011, 121 and 211. Figure 1 shows two of these, the 110 and 011 topographs, which exhibit a variety of features including several different subgrains and a network of dislocations labeled "N." The thick dark line extending through the dislocation array is a scratch placed on the surface. Topographs taken before and after the scratch was introduced show that the network was only slightly disturbed and that no dislocations propagated from the immediate vicinity of the scratch. Feature "D" is a "dent" which was present prior to crystal growth as a result of local plastic deformation. The dislocations which formed this "dent" annealed out, and the remaining deformation is elastically accommodated. The gradient of elastic strains about the "dent" leads to unusual contrast of the dislocation network in the 011 topograph: rather than the more usual enhanced diffraction at defects, the radiation is scattered away from the defect leading to a decrease in diffracted intensity relative to the background. This contrast is similar to that observed in elastically bent silicon crystals (Meieran and Blech, 1972) and can be expected from dynamical scattering theory (Hart, 1981).

The network of dislocations is labeled in Fig. 2 with the Burgers vectors determined using the above contrast criteria. Residual contrast, however, proved to be a major problem in identifying a consistent set of Burgers vectors. Optical densitometry was required to conclusively determine whether the images were in contrast or in residual contrast. The difference in transmitted intensity between the dislocation image and the

background, normalized relative to the intensity transmitted through the unexposed portions of the emulsion, was used as a parameter to determine whether the image was in contrast or was exhibiting residual contrast. As can be seen in Fig. 2, the network's dislocation Burgers vectors consist of $\langle 111\rangle$, $\langle 100\rangle$, and $\langle 110\rangle$ types. While it is energetically favorable for two $a/2\langle 111\rangle$ slip dislocations to combine to form a single $a\langle 100\rangle$ dislocation, the reaction of two $a/2\langle 111\rangle$ dislocations to form a single $a\langle 110\rangle$ is energetically unfavorable. It appears, however, that during the high temperature anneal both $a\langle 100\rangle$ and $a\langle 110\rangle$ dislocation segments form in the network. The fractions of Burgers vectors of each type are 60% $a/2\langle 111\rangle$, 25% $a\langle 100\rangle$ and 15% $a\langle 110\rangle$. Networks comprised of the same types of dislocations were observed by Dingley and Hale (1966) using TEM in Fe, Fe - 0.75%Mn and 2 1/4%Cr - 1%Mo steel. They reported similar distributions of Burgers vector types with 60% $a/2\langle 111\rangle$, 20% $a\langle 110\rangle$ and 20% $a\langle 100\rangle$ types.

Nearly perfect niobium crystals have been reported before, but apparently the perfection of strain-annealed and recrystallized ribbon specimens has not been previously examined. Reed, Guberman and Baldwin (1967) have prepared niobium crystals from the melt which contained widespread networks of dislocations and dislocation densities approaching 10^2 cm/cm^3. Using a crucible-less pulling method, Naramoto (1973) has also grown excellent niobium crystals, containing long dipoles of $a/2\langle 111\rangle$ edge dislocations, long segments of $a/2\langle 111\rangle$ screw types, short $a\langle 101\rangle$ segments and small $a/2\langle 111\rangle$ prismatic

loops and helices. The presence of the prismatic loops and helices was ascribed to vacancy precipitation during growth. The absence of loops and helices in the present study could be due to a variety of factors. The small specimen thickness and the availability of the surface as a defect sink would tend to limit vacancy loop formation. The relatively good vacuum used would also minimize oxide formation during cooling and would limit the subsequent vacancy injection at moderate temperatures. Studies in which loops and helices were observed in molybdenum (Becker and Pegel, 1969) and in niobium (Naramoto, 1978 and Zedler, 1967) were characterized by slower cooling rates and poorer vacuums (10^{-5}-10^{-6} torr).

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FIGURE CAPTIONS

Fig. 1. Lang topographs of a niobium crystal with surface normal approximately [$\bar{1}$10]. a) Subgrains "A" and "B," dislocation network "N," scratch "S" and dent "D" are seen with g = [110]. b & c) Contrast of the dislocations in the network differs for g = [110] and g = [011], respectively, due to the influence of bending from the nearby dent "D."

Fig. 2. Dislocation Burgers vectors for a network "N" in a niobium crystal with surface normal approximately [$\bar{1}$10]. Different line segments, single, double, dashed and dotted, represent dislocation lines with a/2⟨111⟩, a⟨110⟩, a⟨100⟩ and undetermined Burgers vectors, respectively. The direction of Burgers vectors which lie in the plane of the foil are indicated by arrows.

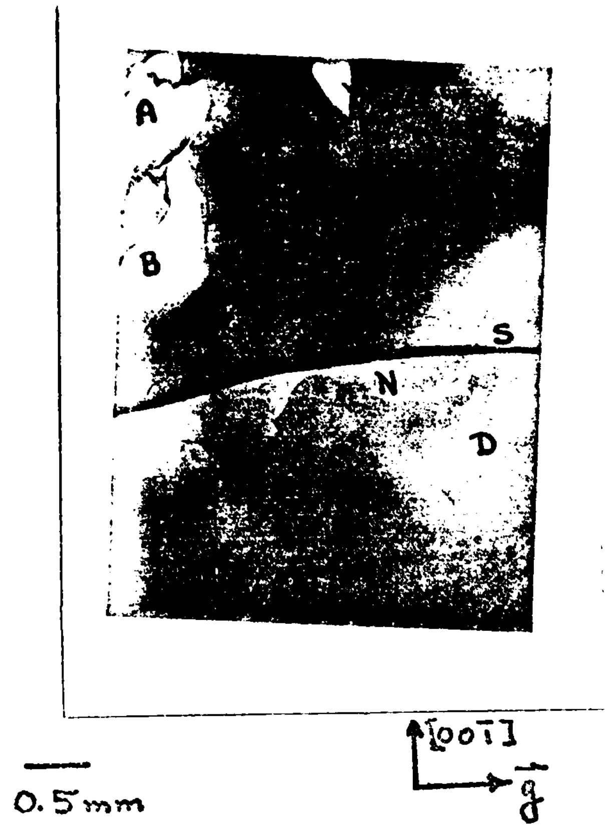

fig 1a

0.5 mm

$[00\bar{1}]$

$\vec{g}$

fig 16

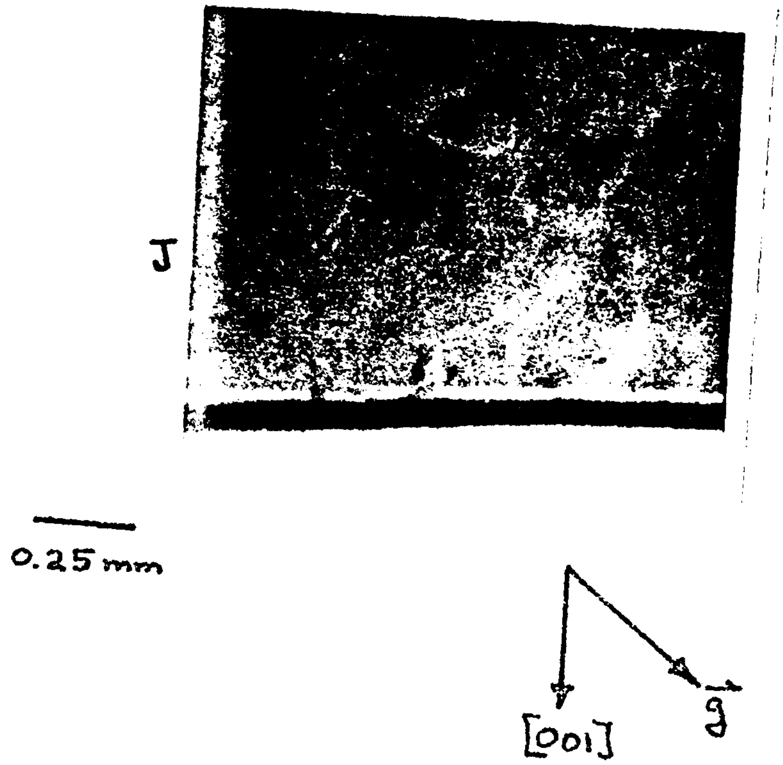

fig 1c

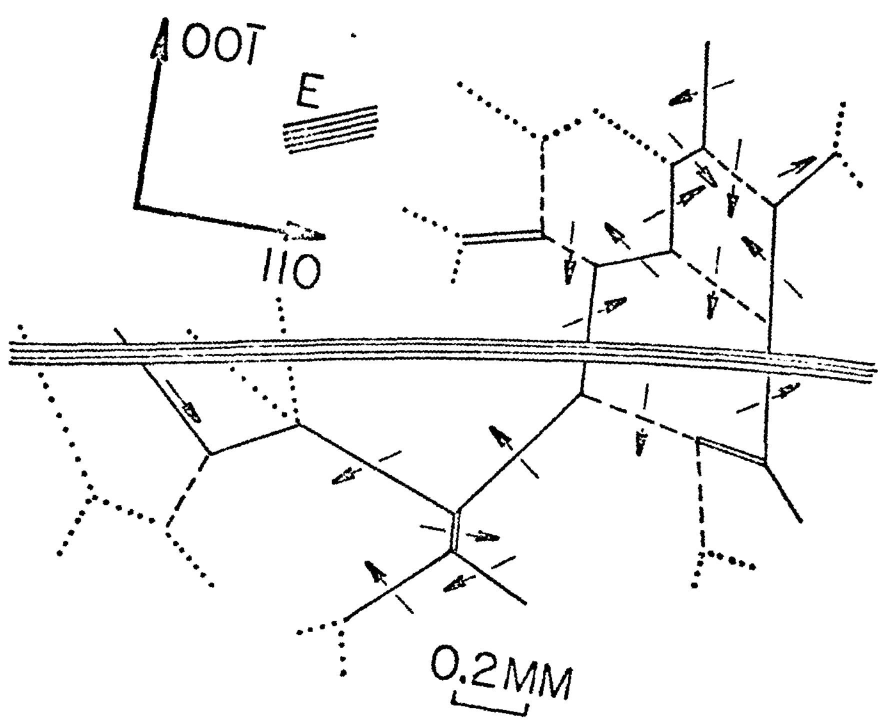

fig 2

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